# PATENT SPECIFICATION

613,041

*Convention Date (United States of America): Nov. 30, 1944.*

*Application Date (in United Kingdom): Oct. 30, 1945.* *No. 28771/45.*

*Complete Specification Accepted: Nov. 22, 1948.*

Index at acceptance:—**Classes 72,** B1b3x; **82(i),** A1(c: d: e: f), A4f, A7, A8(a: i: j: k: m: o: q: r: y), A8z(3: 5: 12: 13), A9a; **and 83(i),** F16bx.

COMPLETE SPECIFICATION

## Improvements in Methods of Producing Alloy Bodies

We, AMERICAN ELECTRO METAL CORPORATION, a corporation registered under the laws of the State of Maryland, United States of America, of 320 Yonkers Avenue, Yonkers, New York, United States of America, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to a method of manufacturing shaped bodies of alloys or aggregates of predetermined constitution, i.e. comprising desired constituents in predetermined proportion. According to the invention, the method of producing dense, shaped metallic bodies comprises infiltrating one or more molten alloys into a shaped porous alloy matrix containing the same constituents as those contained in the infiltrant but in a different proportion and having a higher melting temperature than that of the infiltrant, the infiltrating temperature being between the melting temperatures of the infiltrant and matrix. If, for instance, a shaped body of ferrous alloy of desired composition is to be produced, at least two different ferrous alloys are selected the constituents of which are, besides unavoidable or usual impurities in small amounts, the same as those of the ferrous alloy to be manufactured whereas their proportions are such that the melting temperature of one alloy is considerably higher than that of the other or others. The alloying constituents of the ferrous alloy may comprise combined carbon or graphite and/or various metals.

In carrying the method of the invention into effect, a body permeated by inter-communicating pores is formed from the alloy of higher melting point. A quantity of the other alloy or allovs of considerably lower melting point sufficient at least to fill the pores of that body is brought in contact with the latter, and they are heated so that the lower melting point alloy, or alloys, melts in contact with the body comprising the higher melting alloy and is infiltrated or imbibed into the latter so that on cooling a dense body results. The structure of the body thus obtained is determined by the kinds and proportions of the constituents of the thus combined alloys, and the thermal conditions of the infiltration process and subsequent cooling. The structure of the phase or phases present in the final body can be changed further by treatments of the final body, such as heat treatments of the nature of homogenizing and annealing.

The invention permits the production of shaped bodies of desired composition and proportion of the constituents in a less expensive manner than heretofore, and particularly from relatively inexpensive initial raw materials. The invention further permits the production of relatively large, dense and shaped bodies of metal alloy of desired composition by an infiltration process, in which economical and, in particular, low pressures are used.

Taking as a first example the non-ferrous copper-zinc system, the constitution diagram of which is well known. According to the invention at least two component alloys are chosen from the system so that their melting points differ considerably. Thus, for instance, for the manufacture of a shaped and dense body comprising 70% copper and 30% zinc, a higher melting point alloy comprising 95% copper and 5% (by weight) zinc is selected, having a melting point of about 1050° C.. and a lower meltintg point alloy comprising 50% copper and 50% zinc which melts at about 875° C.

A body or matrix of desired shape is presesd in a die from fragments or powder of higher melting point alloy, the body to have a porosity of about 45%; this means that 55% of the volume of the

shape is occupied by the higher melting point copper-zinc alloy, whereas 45% of that volume is constituted by intercommunicating pores. Preferably small scrap fragments, such as chips, turnings, borings, shavings, are used which are purified and deoxidized, if necessary. If the size of the body is large enough, the fragments can be used as available in the market. If comparatively small sized bodies are to be made, the fragments can be crushed or comminuted to desired smaller particle size by ball milling for a few hours which is inexpensive and does not cause appreciable wear of the mills.

A quantity of the lower melting point copper-zinc alloy sufficient at least to fill the pores of the matrix, is brought into contact with the latter, and both are heated to a temperature of 875° C. or slightly above, so that the lower melting point alloy melts and is infiltrated into the porous matrix which stays solid at that temperature during the short period of infiltration. If the lower melting point alloy is positioned below the porous matrix, its melt is absorbed by the matrix by capillary action; if it is positioned on top of the porous matrix, its melt is infiltrated into the matrix by action of gravity as well as capillary action.

The matrix and melt therein are cooled thereafter so that the latent heat of fusion is abstracted from the melt and it solidifies, whereupon by continued abstraction of heat the matrix and frozen melt therein are further cooled.

If a mere aggregate is desired, cooling can proceed rapidly, for instance in the air or in a protective atmosphere of a cooling chamber of a horizontal or inclined furnace, for instance a continuous furnace. Such furnaces can include a heating zone in which the matrix and the lower melting point alloy in contact therewith are heated to or slightly above the melting temperature of the lower melting point alloy and held at that temperature until the alloy is melted and absorbed by the matrix, and a subsequent cooling zone in which the melt is frozen and the body cooled further. The porous matrix and lower melting point alloy may also be transported downwardly through a vertical furnace space which comprises an upper heating zone in which the matrix and infiltrant are heated to or slightly above the melting temperature of the infiltrant and the latter is melted and absorbed by the matrix, a first cooling zone below the heating zone in which heat is abstracted gradually from the matrix and melt so that the latter is frozen gradually from the bottom toward the top, and another cooling zone below the first one in which the matrix and the solidified infiltrant therein are cooled further or subjected to a heat treatment of a nature described hereinafter.

Whereas upon infiltration followed by cooling an agglomerated or composite body results, cooling below the melting point of the infiltrant can be conducted so that diffusion to desired extent of the zinc from the infiltrant into the matrix results, particularly until a uniform concentration of the zinc in both the infiltrant and the matrix is obtained. In the latter case a homogeneous alpha-phased alloy of the two constituents, copper and zinc, in the predetermined proportion results.

From the above the many advantages of the invention can be realized. Scrap of brass of high copper concentration is being utilized for the matrix; the latter can be pressed to the porous shape under low pressures between about 2 to 10 tons per square inch, in relatively inexpensive presses and with least wear of the dies. Scrap of brass high in zinc can be used as infiltrant and a dense body of desired final shape is obtained by a heat treatment the maximum temperatures of which are well below the temperatures at which the resulting alloy (brass comprising 30% zinc and melting at about 905° C.) can be cast. Apart from the economy as to raw materials, equipment and heat treatment, the further advantage is obtained that a dense body practically ready for use results and subsequent shaping by machining or coining under high pressure is dispensed with. By preshaping a porous matrix under comparatively low pressure, e.g. up to about 10 tons per square inch, and consolidating the matrix to a dense body by infiltrating an alloy comprising the same constituents as the matrix but in such proportion that it melts at a temperature at which the matrix stays solid, high pressures and heavy, expensive presses are avoided. The density of the resulting body nevertheless exceeds considerably that of one coined from powdered or granular metal by highest pressures heretofore used.

If the difference between the melting temperatures of the matrix and infiltrant alloys is relatively small, energetic cooling should be applied immediately after infiltration is completed so as to prevent the matrix from softening and losing its shape, or to prevent diffusion of zinc from the melted infiltrant into the solid matrix material to an extent that the melting point of the matrix material is reduced and the matrix softens and loses its shape.

Instead of using two alloys from the copper-zinc system, there can be used three alloys. In order to obtain a dense body comprising for instance about 64% copper and 36% zinc, a porous body is shaped under low pressure from about 40% (by weight of the final body) of scrap or powdered scrap of an alloy comprising 90% copper and 10% zinc, and heated in contact with about 40% (by weight of the final body) of scrap of an alloy comprising 40% copper and 60% zinc, having a melting point of about 840° C., and about 20% (by weight of the final body) of scrap of a Muntz metal comprising 60% copper and 40% zinc, melting at about 905° C. Upon rather rapid heating to and at a temperature of about 905° C., the two alloys of higher zinc concentration melt while the shaped porous body stays solid and absorbs the molten alloys. After infiltration is completed, the body and melt absorbed therein (which freezes at about 865° C. corresponding to its total zinc content of about 54%) are chilled energetically so as to cool them to that freezing temperature, abstract the latent heat of fusion of the infiltrated melt and cause it to freeze before diffusion of zinc from the melt into the solid material progresses so far that the latter's melting point is reduced and it softens. After the absorbed melt has been frozen, further abstraction of heat can be conducted in various manners. If a mere aggregate is desired, cooling can progress rapidly. If homogenisation by diffusion in the solid state is desired, rapid cooling is stopped well below 865° C. but above about 800° C., and this temperature is maintained until diffusion of zinc from any phase of higher zinc concentration into another phase of lower zinc concentration is completed and a shaped, dense body comprising homogeneous alpha-phased brass is obtained. The advantages of the invention are present in that a shaped body ready for use is obtained as in a powder metallurgical process, and scrap can be used of some or all of the component alloys. While scrap for shaping the porous matrix should be crushed or powdered if bodies of small size are to be produced, scrap of the other two alloys of higher zinc concentration can be used in fragments as available in the market.

Compared with known powder metallurgical methods, the invention offers the advantages that low pressures only are required and that changes in analysis due to evaporation of zinc or overstraining during compacting are avoided. Furthermore, only the infiltrant alloy is melted which need not be subjected to an atomisation or powdering step.

Homogenisation of the shaped composite body obtained according to the invention can be accomplished either in the same furnace in which the infiltration process has been performed, or the body, cooled below freezing temperature of the absorbed belt, can be introduced in another heating chamber for homogenisation or annealing to any desired extent. In the latter case any type of continuous resistance or high frequency furnaces can be used for infiltrating successively the lower melting alloy or alloys into a great many shaped bodies, and thereafter a larger batch of them is heat treated in another heating chamber at a homogenising or annealing temperature for a proper length of time. Hence the process according to the invention is advantageous over the direct manufacture of shaped bodies in conventional powder metallurgical processes, first as to cheapness of some or all the raw material used, second as to low pressures for compacting the porous matrix and consequent lower price of the equipment and reduced wear of the dies, third as to the short period of heating at relatively high temperature and consequent possibility of manufacturing a far greater number of shaped bodies in the same furnace in a unit of time and without losses of lower melting alloy by evaporation.

From the above it will be appreciated that the invention is applicable to an alloy system the constitution diagrams of which show an alloy that stays solid at a temperature at which another alloy of the same system but of different portions of its constituents, readily melts, such as for instance the copper-zinc, copper-nickel, nickel-chromium and other binary (and ternary) systems. The higher melting alloy is used for shaping therefrom under low pressure a porous matrix whereas the lower melting alloy from the same system is used as the infiltrant for consolidating and densifying the matrix. By heat treating the composite body below the melting point of the infiltrant, desired structural changes can be accomplished, such as homogenisation to any desired extent, or annealing for the mere removal of stresses resulting for instance from rapid cooling of the composite body.

The invention is particularly advantageous if alloys of sufficiently differing melting points can be taken from the same system of alloys which are in commercial use and of which inexpensive scrap is available.

Turning now to ferrous materials, it should be clear from the above that by the invention shaped bodies resembling

steel or alloy steel can be obtained in a more economical and simple manner than heretofore. Ferrous alloys concerned by the invention comprise iron as a base and one or more alloying constituents in solid solution, or as an eutectic. Thus the alloying constituents may comprise either combined carbon or carbon in the form of graphite, and/or metallic or non-metallic matter, such as for instance silicon, manganese, chromium, sulphur, aluminium, nickel, cobalt, tungsten, molybdenum, singly or in desired mixture. Again from the constitution diagram of a system comprising iron and one or more of the alloying constituents, at least two alloys are selected in which the proportions of the constituents differ so that one alloy stays solid at the temperature at which the other selected alloy or alloys readily melt. From the alloy of highest melting point a porous matrix is shaped under relatively low pressure, and the other alloy or alloys of considerably lower melting point is or are infiltrated into that matrix, and the whole is chilled so that the infiltrated melt is frozen before the porous matrix softens and loses its shape. If structures other than mere agglomerates are desired, one or more heat treatments below the melting temperature of the infiltrant or the composite body are subsequently applied, for instance for the purpose of homogenization to any desired extent, producing a desired structural change, annealing or normalizing.

Taking as a first example the manufacture of a shaped ferrous body comprising 1.5% carbon from low carbon steel comprising .5 carbon and melting at about 1485° C., and cast iron comprising 4% carbon (disregarding unavoidable impurities) and melting at about 1260° C. Scrap of both kinds of ferrous alloys are available in the market in large quantities at low price. If a relatively large body, such as for instance a gear of 6″ to 12″ diameter and 2″ to 3″ thickness is to be produced, turnings, shavings, borings and other kinds of steel scrap of the composition stated and which may be purified and deoxidized, if necessary, can either be used as such for pressing the porous matrix or after the scrap has been crushed to smaller fragments. If smaller sizes are to be compacted, the scrap should be powdered. A shaped and porous body is compacted therefrom having a porosity of 29%. i.e. only 71% of the shape is occupied by that steel leaving interconnecting pores constituting 29% of that volume. For compacting such shape, pressures of about 10 to 30 tons per square inch ordinarily suffice, which are commercially obtainable by relatively light presses and result in insignificant wear of the die. Cast iron scrap, purified and deoxidized, if necessary, and the quantity of which suffices to fill the pores of the porous matrix, is heated in contact with the latter to 1260° C. or slightly higher, and is thereby melted while the matric stays solid. The melt is absorbed in the matrix by capillary action if the cast iron was positioned below the matrix in a proper receptacle of highly refractory material. The melt is infiltrated in the matrix by gravity and capillary action if the cast iron was positioned on top of the porous matrix. The pores of the matrix are large enough to secure rapid infiltration. After infiltration is completed, the matrix with the melt absorbed therein is chilled so as to freeze the infiltrant, and the solid composite body thus obtained is further cooled in any desired manner, rapidly or slowly, depending on whether diffusion of carbon from the frozen cast iron into the matrix material is desired. The composite body may also be cooled in air. Any known type of furnace and method of infiltration can be used, particularly as described hereinbefore with respect to non-ferrous alloys. The rapidly cooled composite body can be removed from the furnace and introduced into a heat treating chamber in order to anneal or to homogenise it, i.e. cause diffusion of carbon from the infiltrant of higher carbon concentration into the matrix of lower carbon concentration, for instance until an equilibrium is obtained. On proper cooling any desired phase structure of the body can be attained.

In the above example a composite body of an average carbon content of 1.5% results. By heat treating the body at temperatures between 900° C. and 1100° C. for a sufficient period of time, homogenisation by diffusion of carbon in the solid austenitic state can be accomplished. On slow cooling the thus homogenised body below about 723° C., a phase structure resembling that of hypereutectic steel results.

As another example a shaped and dense body of .7% over-all carbon content was made of 80% (by weight of the final body) cold rolled low carbon steel scrap and 20% (by weight of the final body) cast iron shot, containing about 3.5% carbon. The cold rolled steel machinings were milled to about 60 mesh particle size and two porous cylindrical compacts or matrices of ¾″ diameter and ¾″ height pressed therefrom at a pressure of 25 tons per square inch. The resulting compacts had a porosity of about 20% and were placed in the cavity of a highly refrac-

tory mold. A quantity of cast iron shot, sufficient to fill upon infiltration the interconnecting pores of one compact, was positioned on top of it in the cavity of the mold and heated in contact with it by moving them continuously downwardly at a speed of ½" per minute through a vertical furnace space in which a proper protective atmosphere was maintained by leading therethrough as dry as possible hydrogen. The temperature in the furnace space was carefully controlled to raise it to 1200° C. and thereafter to gradually lower it to below 725° C. The porous matrix and infiltrant were kept at a temperature of above 1150° C. for about 15 minutes and thereby the infiltrant was melted and absorbed by the matrix. After solidification and slow cooling, the compact with the cast iron infiltrated therein was cut, etched and examined at 230 magnification and 860 magnification. The examination revealed uniform infiltration of the infiltrant and diffusion to large extent of the excess carbon of the infiltrant into the low carbon matrix as exhibited by the large areas of pearlite in the network of ferrite. Micro-hardness impessions were made and indicated that the pearlite area if of greater hardness; the average hardness was 180 Brinell.

The second compact was heated in contact with the same kind of cast iron (positioned on top of it) in a batch-type muffle furnace to a temperature of about 1200° C. in the presence of as dry as possible hydrogen: a temperature above 1150° C. was maintained for 15 minutes whereby melting and infiltration of the cast iron into the matrix was accomplished. The specimen was slowly cooled below 725° C.; after cooling to room temperature, it was cut, etched and examined at 230 magnification. It displays far progressed diffusion of carbon, a uniform structure of pearlite areas embedded in a network of ferrite which frequently shows, however, minute pores and specks of cementite. The average hardness was 139 Brinell.

In both cases the base metal skeleton or matrix retained its initial contour and therefore determined the shape of the final body.

In another experiment spongy ferritic iron obtained by reduction of scrap iron and comprising between .05% and 0.1% carbon, was powdered to pass a 100 mesh screen and pressed into two cylindrical shapes of ¾" diameter and ¾" height at a pressure well below 25 tons per square inch so as to exhibit 20% porosity. One compact was infiltrated in the same manner as described with reference to the foregoing experiments, with the same kind of cast iron scrap by lowering the matrix with infiltrant on top through the vertical furnace space, while the other compact with the same kind of infiltrant on top was heated under the same conditions in the stationary furnace. The average carbon content of the slowly cooled bodies was little below .7% and they exhibited the basic structure of hypo-eutectoid steel with pearlite batches embedded in a ferritic matrix.

The specimens proved that sufficiently porous briquets of soft powdered iron or powdered scrap of low carbon steel can be conveniently infiltrated with cast iron, which on homogenisation yield synthetic structures resembling steel or alloy steel By the choice of cast iron of proper carbon content as the infiltrant and of ferritic iron or steel of considerably lower carbon content and therefore higher melting point, for the porous matrix, final bodies can be obtained by infitration the over-all carbon content of which answers a given specification, the shape of which is determined by that of the compacted porous matrix, and the phase structure of which depends on the mode of cooling and heat treating. Articles of considerable size can be compacted in large hydraulic presses; considering for instance the example given above of a gear of 12" diameter and 2" to 3" thickness, obtained by infiltrating cast iron into a shaped porous matrix of low carbon steel, calculation shows that an area of about 110 square inches is to be pressed which requires, with 25 tons per square inch, a hydraulic press in which less than 3000 tons total force can be produced. These are pressures which can be produced in hydraulic presses of customary designs whereas production of dense alloy bodies of similar sizes and of same composition in known powder metallurgical processes require compacting pressures up to 100 tons per square inch, resulting in rapid wear of the dies, and more expensive presses having at least double the press capacity of those needed according to the invention. For smaller sizes and powders requiring, according to the invention, compacting pressures between 5 and 25 tons per square inch, quick acting mechanical presses can be used which produce great quantities per time unit. Due to the great affinity between the matrix material and that of the infiltrant, alloying by diffusion in the solid austenitic state can always be obtained, either during slow cooling or in a subsequent homogenisation heat treatment in a heating chamber. Thereby any desired steel structure can be obtained in a highly synthetic manner.

The use of a vertical furnace through which the matrix with the infiltrant is passed resulted in very dense specimens; in the specimen obtained in a stationary furnace, minute pores appear.

It should be understood that the invention is not limited to the exemplifications hereinbefore described but is to be derived in its broadest aspects from the appended claims.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. The method of producing a dense, shaped metallic body which comprises infiltrating one or more molten alloys into a shaped porous alloy matrix containing the same constituents as those contained in the infiltrant but in a different proportion and having a higher melting temperature than that of the infiltrant, the infiltrating temperature being between the melting temperatures of the infiltrant and matrix.

2. A method as claimed in Claim 1, characterized by heat treating the infiltrated matrix to diffuse the constituents of the alloys uniformly to form a single homogeneous single-phased alloy.

3. A method as claimed in Claim 1 or 2, characterized in that the infiltrated matrix is heat treated by control of its cooling rate from the temperature of infiltration.

4. The method of producing a dense shaped metallic body substantially as hereinbefore described.

5. A dense shaped metallic body produced by a method claimed in any preceding Claim.

Dated this 30th day of October, 1945.

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Leamington Spa: Printed for His Majesty's Stationery Office, by the Courier Press.—1948.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies, price 2s. 0d. each (inland) 2s. 1d. (abroad) may be obtained.